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Raman / Bench Scale Polymerization of Ethylene

Purpose: To determine whether or not a Raman filter is needed on a simulated reactor containing polymer and if so, to determine whether or not the filter will provide an acceptable spectrum.

See 36556-25, J.B. Ashew

Time	File	
11:50	Slurry 1	30 sec acquisition, no filter, IC4 + PE fluff
	Slurry 2	60 sec " " " " "
	Slurry 3	30 sec acquisition, no filter, " " "
11:15	Slurry Filter 30 sec	" " " with filter 60°C, 112 psig
	SLF0001	60 sec acquisition
11:25	SLF0001	60 sec, 60°C, 112 psig, 700 rpm, Ti + SST support polyethylene
	SLF0002	repeat of 1
	SLF0003	Go to 73°C
	SLF0004	
	SLF0005	73°C, 124 psig
	6	80°C, 130 psig
	7	" "
	8	Transition/heat up to 90°C.
	9	90.2°C, 224 psig
	10	90.2°C, 224 psig
	11	Transition to 100°C
	12	100.7, 275 psig
	13	100.1, 273 psig
	14	104.4, 295 psig
	15	105.3, 305 psig
	16	105.1, 301 psig
	17	60.2, 113 psig
	18	Added ethylene to get total reactor pressure = 200 psig
	19	60°C, 190 psig (C ₂ band forward) OK/OK 113 psig IC4 + 87 psig C ₂
	20	60°C, 198 psig
	21	Group to 73°C on this scan.
	22	75°C, 210 psig
	23	↓ ↓ ±4°C fluctuations
	24	
	25	76°C, 210 psig
	26	71.3°C, 240 psig

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Roman / Bruch Scale Polymerizer at 700 G

Purpose: To Return more Data using a Roman Polymerizer
 The Reactor contains 450 grams Polymer & 2 L of ICH
 + stirring at 700 RPM.

8:29 - started.

SLFA - Scans

2 L ICH Polymer 450 grams RPM 700

1	60.0	115	Psy	
2	60.0	115	Psy	
3	83.6	119.6	Psy	Rx Trying to control Temp.
4	80.0	183.	Psy	OK
5	80.0	183.	Psy	OK
6	102.5	299	Psy	Rx Trying to control Temp.
7	100.1	297.6	Psy	OK
8	100.0	297.7	Psy	OK
9	100.0	361	Psy	Add C ₂ H ₅ Br, Rx Trying to control Temp.
10	100.0	361	Psy	OK
11	100.0	362	Psy	OK
12	78.0	270	Psy	Rx Trying to control Temp.
13	85.0	273	Psy	Rx Trying to control Temp.
14	81.4	284	Psy	Need to add C ₂
15	80.2	270	Psy	OK
16	80.0	271	Psy	OK
17	64.0	-	-	Rx Trying to control Temp.
18	59.8	196	Psy	Rx Trying to control Temp.
19	60.0	186	Psy	Need C ₂
20	60.0	200	Psy	Rx Trying to control Temp.
21	60.0	197	Psy	"
22	60.0	197	Psy	Done.

500 C₂H₅Br added for 878Scans 1 thru 22 C₂

was maintained set Psy

for 860C, 80C, 100C

115 Psy, 183 Psy, 299 Psy

+ 878 Psy, 186 Psy, 270 Psy

202 Psy, 270 Psy, 364 Psy

Change to Gas Phase Probe

Scan 23 out side light (Room) Bad Spectrum

24 59.7 186 Psy Gas Phase

25 60.0 182 Psy " "

Re-Adjust Filter on - Change to Liquid Phase Probe

Change C₂H₅Br to 250 Torr. Rx Try. at 60C stir 700 RPM

10:48

SLFB - Scans

1	60.0	245	Psy	
2	60.0	251	Psy	OK
3	60.0	245	Psy	OK
4	82.0	383	Psy	Rx Trying to control Temp.
5	77.0	287	Psy	" "
6	84.0	340	Psy	" "
7	78.0	329	Psy	
8	94.0	422	Psy	Rx Trying to control Temp.
9	100.0	400	Psy	"
10	103.0	410	Psy	"
11	91.0	377	Psy	Temp Cycle
12	103	512	Psy	"

Stop for hours

Cooling Rate 1.0 C/hr

Continued From Page 33 325-34				Stand by back from Lunch.	
P. 1 hr	SLFBZ 000.1	- 66.0°	338 Psig	Add 2° Psig to set 328 Psig.	
IC4	Scan-2	- 64.0°	355 Psig	Temp not controlling. Real good up/down.	
	2	- 57.0°	269 Psig	OK.	
	4	- 55.7°	283 Psig	Rise Temp to 60°	
RAM 700	5	- 87.0°	98.0 Psig		
	6	- 77.0°	333 Psig		
	7	- 60.0°	444 Psig		
	8	- 83.1°	358 Psig	Psig is up + Down?	
	9	- 85.8°	450 Psig	Rise Temp to 100°	
1 Temp	10	- 100.0°	569 Psig		
	11	- 42.0°	425 Psig	← Temp of Rx was 104°	
2 Temp	12	- 104.0°	569 Psig	Up + Down Temp.	
	13	- 45.0°	445 Psig	" " "	
	14	- 96.0°	517 Psig	" " "	
	15	- 105.0°	475 Psig		
3 Temp	16	- 72.0°	288 Psig	Cooling to 60° →	
	17	- 58.0°	305 Psig		
	18	- 62.0°	299 Psig		
4 Temp	19	- 55.0°	236 Psig		
5 Temp	20	- 64.0°	280 Psig		
	21	- 72.0°	332 Psig	Add 50 grams to Rx of H ₂ O	
	22	- 83.0°	279 Psig	Value open to Rx	
Temp	stop to			Flare Rx note-2	
Temp				(To open to Inspect Probe)	
Psig	SLFBZ 000 -1	3.7°	214 Psig	Flare Rx for 20 min. Pressured up with	
	"			N ₂ to 200 Psig	
	2	0.1°	0 Psig	Stirring on, Polymer, vapors	
	"			NO Pressure open to Flare	
	3	0.4°	0	Stirrer Stop	
18:30	SLFBZ 000 -	clean out Rx Polymer		Purge Rx with N ₂	
	Scan 21	30.0°	53 Psig	N ₂ Flow thru Rx	
	Purge to Substrate	30°	83 Psig	2) 2 IC4 + stirring 700 RPM	
	Stirrer on at 20	30°	72 Psig	with Filter + no Polymer	
	"	30°	72 Psig		
	Add 90 grams of H ₂ O	30°	72 Psig	70 grams H ₂ O added	
	"	30°	72 Psig		
	Stop Down Time to 50 min			Flare Rx over night	
	Note: When I clean Rx out of Polymer the Polymer had stuck to the bottom of Rx + some of the wall. (the Probes were clean.)				